

TITLEMETHOD FOR REDUCING THE PARTIAL PRESSURE  
OF UNDESIREG GASES IN A SMALL VACUUM VESSEL

5

This application claims the benefit of U.S. Provisional Application No. 60/448,852, filed on February 20, 2003, which is incorporated in its entirety as a part hereof for all purposes.

10

Field of the Invention

This invention relates to a method for reducing the partial pressure of undesired gases in a small vacuum vessel that comprises using a getter pump as an integral part of the small vacuum vessel.

15

Background of the Invention

Vacuum vessels are used in a wide range of applications in which it is advantageous or necessary to have electronic circuits, sensors or other devices located in a vacuum either to maintain a given temperature or to improve reliability, e.g., to provide corrosion resistance.

25

The discovery and use of high temperature superconductor (HTS) materials that superconduct at temperatures of 77K or higher have increased the need for small vacuum vessels, which permit operation at cryogenic temperatures. They have been used in various industrial, medical, research and military applications. As a result of the growth in the telecommunications industry, one of the fastest growing commercial applications has been in the area of electronics and associated microwave engineering. In this area, an essential part of many devices is the filter element. HTS filters have significant

30

35

advantages in insertion loss and selectivity due to the extremely low radio frequency (RF) loss in HTS materials. Amplifiers and other circuit components, as well as the HTS filters, can be contained within a  
5 small vacuum vessel. One such application is a cryogenic receiver front-end in which cryoelectronic components such as RF filters and amplifiers are contained within the small vacuum vessel.

10 The small vacuum vessel is evacuated to a high vacuum in order to more readily maintain the cryoelectronic components at cryogenic temperatures. Maintaining this high vacuum is critical to the performance of the cryoelectronic components contained  
15 in the small vacuum vessel.

Desorption of gases adsorbed on the inner surfaces of the small vacuum vessel and on the surfaces of its contents, i.e., outgassing, is therefore important.  
20 Typical outgassing practice is to subject the small vacuum vessel to a bakeout step, i.e., maintaining the small vacuum vessel and its contents at a high temperature while under a high vacuum , i.e.,  $10^{-6}$ - $10^{-7}$  mbar. The materials used in the construction of the  
25 small vacuum vessel and its contents and the temperature to which they can be subjected must be considered when deciding on the temperature used for this bakeout. This often dictates the use of lower temperatures. Longer bakeout times are needed at these  
30 lower temperatures to provide the necessary outgassing. The small vacuum vessel and its contents are typically heated to a temperature in excess of 50°C for a length of time up to a week. Even with bakeout temperatures on the order of 100°C, bakeout times can be on the  
35 order of 48 hours.

A getter pump is usually provided in the small vacuum vessel to adsorb gases that are present as a

result of any additional outgassing that occurs after sealing the vacuum vessel or as a result of small leaks. The getter pump is activated by heating to remove any oxide and nitride coatings. Getter pump  
5 activation is carried out before sealing the small vacuum vessel. This process requires a time on the order of 4 hours for the small vacuum vessel to cool down after the bakeout, and a time on the order of an hour for the pulse activation of the internal  
10 electrical heaters of the getter pump to avoid overheating the internal parts of the small vacuum vessel. The small vacuum vessel can then be sealed.

The specific times actually required for the steps  
15 indicated above depend on the size and contents of the small vacuum vessel. However, the typical times given are indicative of the considerable time and cost to the production of the small vacuum vessel. In addition, this outgassing process requires the purchase and  
20 maintenance of a high vacuum system dedicated to the bakeout cycle and equipment needed to bakeout the small vacuum vessel.

An object of the present invention is to provide  
25 an improved method for more efficiently reducing the partial pressure of undesired gases in a small vacuum vessel.

### 30 Summary of the Invention

This invention relates to a method for reducing the partial pressure of undesired gases in a small vacuum vessel that comprises the use of a getter pump as an integral part of the small vacuum vessel. The  
35 method comprises (a) providing a getter pump as an integral part of the small vacuum vessel, wherein the getter pump has sufficient sorption capacity to adsorb undesired gases; (b) evacuating the vacuum vessel;

(c) activating the getter pump; and (d) sealing the vacuum vessel.

5 Preferably the getter pump is in the form of an appendage getter pump that is attached to and made a part of the small vacuum vessel.

10 Yet another embodiment of this invention is a method for reducing the partial pressure of undesired gases in a small vacuum vessel with a vacuum volume of about 1 liter or less, said small vacuum vessel having undergone an incomplete outgassing bakeout step, by providing a getter pump as an integral part of said small vacuum vessel, wherein said getter pump has  
15 sufficient sorption capacity to adsorb remaining undesired gasses.

20 Yet another embodiment of this invention is a small vacuum vessel with a vacuum volume of about 1 liter or less, that includes a getter pump as an integral part of said small vacuum vessel, wherein said getter pump has sufficient sorption capacity to adsorb undesired gases without the necessity of an outgassing  
25 bakeout step.

Yet another embodiment of this invention is a method for reducing an increase in temperature experienced by a vacuum vessel, having a vacuum volume of about 1 liter or less, from the reduction of the  
30 partial pressure of undesired gases in the vacuum vessel by (a) providing as an integral part of the vacuum vessel a getter pump that has sufficient sorption capacity to remove undesired gases from the vacuum vessel; and (b) before sealing the vacuum  
35 vessel, heating the vacuum vessel only to the extent required to activate the getter pump.

## Detailed Description of the Preferred Embodiments

The present invention provides a method for reducing the partial pressure of undesired gases in a small vacuum vessel containing a getter pump as an integral part of the small vacuum vessel. No bakeout is necessary with the instant method and the time interval between the beginning of evacuation of the small vacuum vessel to sealing of the small vacuum vessel is reduced to the order of an hour instead of the typical 4-5 days of the current practice. The work of the getter pump in removing contaminants is thus performed at the temperature and pressure at which the pressure vessel is operated (i.e. the temperature at which it is placed and kept in service), and decontamination is thus performed in this invention in the absence of the elevated temperatures typical of previous bakeout methods.

A small vacuum vessel is a vacuum vessel with a vacuum volume of about 1 liter or less. Undesired gases are gases adsorbed on the inner surface area of the small vacuum vessel and on the surfaces of its contents and subject to outgassing, as well as gases present as a result of small leaks.

The getter pump is an integral part of the small vacuum vessel and must be chosen appropriately to have sufficient sorption capacity to adsorb the undesired gases.

The quantity of undesired gases as a result of outgassing can be estimated based on the inner surface area of the small vacuum vessel and the surface area of its contents and the particular materials used in the construction of the small vacuum vessel and its contents. Typically the volume of adsorbed gases is equivalent to a few monolayers coverage of the internal surface. Allowance must be made for the varying degree

to which the different components or materials used  
tend to outgas. The quantity of undesired gases as a  
result of small leaks can be estimated on the basis of  
degree of leakage that is to be provided for. A getter  
5 pump is then chosen with a sorption capacity of several  
times, for example at least 3-5 times, that estimated  
to be needed in order to provide a safe margin of  
design. This getter pump can then be tested in a small  
vacuum vessel to determine if the estimate was correct  
10 and performance is as expected. If not, a getter pump  
with even greater sorption capacity, for example at  
least an additional 2-3 times more sorption capacity,  
can be used and tested. When the expected performance  
has been obtained, such a getter pump can be used in  
15 all similar small vacuum vessels produced. The use of  
a getter pump with the greater sorption capacity as  
described above will enable relaxation of restrictions  
with regard to outgassing placed on materials chosen  
for the construction of the small vacuum vessel and its  
20 contents.

The getter pump can be in the form of pellets of  
non-evaporable getter material contained in an  
appendage that is an integral part of the small vacuum  
25 vessel or in the form of an appendage getter pump  
available commercially. The getter pump is activated  
by heating to remove any oxide or nitride coatings.  
This activation is carried out with the small vacuum  
vessel evacuated but before the sealing of the small  
30 vacuum vessel. This heating can be accomplished by  
passing an electrical current through internal heaters  
provided with the getter pump, which are located inside  
the pressure vessel, but preferably is done by heating  
the appendage to a temperature sufficient to activate  
35 the getter material contained therein by means of an  
external heater outside the pressure vessel designed to  
fit around or encompass the appendage. Care must be  
taken to avoid overheating the sensitive components of

the small vacuum vessel and its contents during activation. The getter pump can be placed sufficiently far from or shielded from the small vacuum vessel contents to protect the contents from a harmfully high temperature when the getter is activated. Careful design and the use of an appendage getter pump can therefore result in a significant reduction in the maximum temperature experienced by the contents compared to that occurring during a typical outgassing bakeout step.

If a shortened incomplete outgassing bakeout step is used and a significant amount of gas is still adsorbed on the inner surface of the small vacuum vessel and the surfaces of its contents, the instant invention provides a method for reducing the partial pressure of remaining undesired gases, i.e., the remaining undesired gases adsorbed on the inner surface of the small vacuum vessel and the surfaces of its contents. In this instance, the getter pump must be chosen appropriately to have sufficient sorption capacity to adsorb these remaining undesired gases.

Typical getter material that can be placed within an appendage are Zr-V-Fe alloys such as the getter pump St 707<sup>TM</sup> available from SAES Getters, S.p.A., Milan, Italy. Preferably, the getter pump is in the form of an appendage getter pump available from SAES Getters USA Inc., Colorado Springs, CO 80906. The appendage getter pump can be bolted to the small vacuum vessel using holes in the flange provided by the vendor or it can be welded to the small vacuum vessel.

The method of the invention for reducing the partial pressure of undesired gases in a small vacuum vessel requires considerably less time than the method currently used and eliminates the need for a bakeout cycle and the time it consumes as well as the need for

an expensive high vacuum system dedicated to the  
bakeout cycle and the need for equipment to bakeout the  
small vacuum vessel.